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STRUCTURAL MATERIALS DIVISION

STRESS-CORROSION CRACKING
OF HIGH-STRENGTH ALLOYS

A Report To

FRANKFORD ARSENAL

Contract DA-04-495-ORD-3069

Report No. 0414-02-3 (Quarterly) / April 1964 / Copy No.

AEROJET GENERAL

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OF HIGH-STRENGTH ALLOYS

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AEROJET-GENERAL CORPORATION
A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

Report No. 0414-02-3

This is the twelfth in a series of quarterly progress reports submitted in partial fulfillment of Contract DA-04-495-ORD-3069. It constitutes the third quarterly progress report for the second 1-year continuation of the original 2-year program.

This report covers the period 1 January through 31 March 1964. It was written by R. B. Setterlund who was supervised by A. Rubin.

AEROJET-GENERAL CORPORATION

Dr. S. Brelant, Manager
Materials Engineering Department
Structural Materials Division

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I. OBJECTIVE

The objectives of the program are outlined below.

A. To study the stress-corrosion characteristics of 18%-nickel maraging steel with respect to compositional variation.

B. To study the effect of environmental temperature on the rate of stress-corrosion cracking in three alloys: 18%-nickel maraging steel, a low-alloy martensitic steel, and a hot-worked die steel.

C. To study the electropotential changes occurring in 18%-nickel maraging steel during stress-corrosion exposure and the effect of applied potential.

D. To evaluate the effectiveness and applicability of surface protection on 18%-nickel maraging steel in preventing stress-corrosion cracking.

II. BACKGROUND

The present program is the second 1-year extension of the original 2-year program on stress-corrosion cracking of high-strength alloys. During the first 2-year study, six alloys were evaluated: Ladish D6AC steel; Type 300M steel; Vascojet 1000 steel; AM 355 cold-worked PH steel; precipitation-hardening 15-7Mo stainless steel; and B120VCA titanium. Significant failures were found to occur with the D6AC, 300M, and Vascojet 1000 steels in tap, distilled, and salt water, as well as in high-humidity environments; the time to failure for each of the three steels was found to decrease with increasing strength.

During the first 1-year continuation program, attention was focused on three new high-strength steels plus one high-strength titanium alloy. These alloys are: 20%-nickel maraging steel; 18%-nickel maraging steel; 9Ni-4Co vacuum melted steel; and 6Al-4V titanium. The titanium alloy showed complete immunity to stress-corrosion failure under all test conditions. Limited susceptibility was

noted for the 9Ni-4Co alloy. High susceptibility was noted with both the 20%- and 18%-nickel maraging steels. Since the original study of maraging steels was started, the 18%-nickel grade has received increased attention in the aerospace industry, and is now of primary interest. The present program is therefore directed to the study of the stress-corrosion behavior of this one alloy, with emphasis on compositional variation, effect of environmental temperature, and study of electropotential changes. It is intended to: first, determine the extent of the stress-corrosion problem in 18%-nickel maraging steel by testing four additional heats; second, compare the susceptibility of maraging steel with conventional ultra-high-strength steels; and third, to investigate further, by electropotential methods, the cause of failure of 18%-nickel maraging steel.

III. TEST PROCEDURES

A. BENT BEAM TEST

The bent-beam test is the primary test method used in the program. Figure 1 shows an insulated bent-beam fixture with test samples mounted. Polycarbonate blocks 7.000 ± 0.001 in. apart, attached to a stainless-steel holder, support the test specimen and insulate it from the holder. Specimens are cut to exact length to give a maximum outer-fiber stress of 75% of the 0.2% offset yield strength. The length-stress relationship is shown in Figure 2.

B. U-BEND TEST

In addition to the bent beam testing, U-bend samples are used to show the effect of elastic stresses combined with plastic deformation. Samples are bent in a special 1 in. radius fixture after heat treatment and cleaning. Samples which were known to have a low ductility were warmed to 100 to 175°F prior to bending.

C. CENTER NOTCH TEST

Figure 3 shows the test specimen configuration used in the accelerated center-notch test. This consists of a 1-3/4 by 8-in. tensile specimen containing a central notch. The notch is produced by a two-step process. First, a 0.06- by 0.57-in. slot is Elox-machined and extended at each end by very narrow Elox-machined notches of 0.001-in. root radii. Second, an extension of these notches is produced by tension-fatigue cycling to obtain fatigue cracks of controlled dimensions.

These center-notched specimens are tested in Baldwin creep-test machines. The desired loads are obtained by dead weight loading applied to a 20-to-1 lever arm. The test environment is applied by cementing a polyethylene cup to the specimen in the area of the notch. These specimens are well adapted to stress-corrosion studies in that crack growth rate, corrosion potential, or corrosion current can be conveniently measured.

D. TEST ENVIRONMENTS

The test environments in this program include those that the results of the previous year's program indicated were the cause of the most rapid failure of maraging steel; these are: continuous immersion in aerated distilled water, continuous immersion in aerated distilled water containing 3% by weight chemically pure sodium chloride and water-saturated air at $140^{\circ}F$. In addition, two new environments are being employed; distilled water at a thermostatically regulated temperature of $120 \pm 0.1^{\circ}F$, and distilled water thermostatically regulated to $160 \pm 0.1^{\circ}F$. All baths are changed every 10 to 14 days.

IV. PROGRAM STATUS

A. COMPOSITIONAL VARIATION

The first objective of the program, the study of compositional variation on stress corrosion susceptibility of 18%-nickel maraging steels, is being fulfilled by the testing of four additional commercial heats in environments known to produce the most rapid failures. The compositional variation of these four heats are shown in Table 1, Group (b). The only element showing wide variation is titanium, which varies from 0.23 to 0.55%. These heats in conjunction with the five listed in Table 1, Group (a), encompass the compositional variations of present day commercial heats of 18% nickel maraging steel. Mechnical properties of the nine heats are shown in Table 2.

Fracture toughness values were determined using the specimen shown in Figure 3. Fracture toughness results show a very definite relationship between critical crack growth energy, G_c*, and yield strength for the nine heats of 18%

*The G_c values tabulated in this report are somewhat higher than shown in previous reports due to the incorporation of the Irwin plastic zone-size correction.

nickel maraging steel tested (Figure 4). These heats show a much greater toughness than low alloy steel, die steel, or 20% nickel maraging steel. Extremes were noted in austenite grain size, Figure 5, but these were found to have little, if any, effect on mechanical properties.

Replicate bent-beam and U-bend specimens of these four heats along with specimens of low-alloy steel and die steel, have been tested in the following environments: an environment containing distilled water and an environment of 3% NaCl in distilled water. The distilled water was found generally to be more severe. These data are tabulated in Tables 3 and 4. The bent beam results in distilled water are shown plotted in Figure 6. While great variations in resistance to stress corrosion cracking exist between the nine heats of 18%-nickel maraging steel, the time to failure generally decreases as the strength increases. Also, 18%-nickel maraging steel is more resistant than the low alloy or hot worked die steels when compared at the same strength level.

B. ENVIRONMENTAL TEMPERATURE

The second objective of the program is the study of the effect of environmental temperature on the rate of stress corrosion cracking in three alloys: 18%-nickel maraging steel, a low-alloy steel, and a hot-worked die steel. To accomplish this we are conducting bent beam and U-bend stress corrosion tests in three environments: distilled water at 120°F, water saturated air at 140°F, and distilled water at 160°F. The results are shown in Tables 3 and 4. Figure 7 shows the effect of temperature on the time-to-failure of U-bend specimens. Temperature has a much greater effect on the failure time of 18%-nickel maraging steel than either the hot-worked die steel or the low alloy steel.

Figure 8 shows a typical stress-corrosion crack in 18%-nickel maraging steel after exposure to 160°F distilled water. Figure 9 shows typical stress-corrosion cracks in the two conventional steels. In all three cases, the cracks were intergranular.

C. ELECTROPOTENTIAL CHANGES

The third objective of the program is to measure the electrochemical changes occurring in 18% nickel maraging steel during stress corrosion exposure and

to determine the effect of applied potential on failure time. An experiment has been conducted on a center-notched specimen of heat 3960502. The test setup is shown in Figure 10. Using this method, the applied stress vs crack potential relationship of Figure 11 was obtained. As the stresses at the crack tip, K, increase as a result of incremental loading of the metal, a potential shift toward the cathodic is noted. A second test of this type will be conducted where the stress, K, will be held constant and the potential recorded during stress corrosion exposure until failure results.

Tests have begun on the effect of applied potential on the stress corrosion cracking of 18%-nickel maraging steel. Figure 12 shows the test setup for this portion of the program. A Duffers potentiostat is utilized to control the potential of a U-bend specimen to a preset value. Two runs on U-bend samples of heat 07868 in 3% NaCl solution have been conducted to date, with the following results:

Potential to Saturated Calomel Cell, v	Current Density, ma/in.^{2}	Corrosion	Stress Corrosion
-1.50	-80 (cathodic)	Not noted	Many cracks after 1 hour
-0.40	None	Mild	Failed after 15 days
+0.50	+123 (anodic)	Severe	Many surface fissures in 2 hours

These runs were intended to produce metallographic specimens of 18%-nickel maraging steel that had failed under anodic and cathodic polarization. The actual mechanism study will involve U-bend specimens of 20%-nickel maraging steel and low alloy steel tempered at 600°F. These specimens had failure times in salt water without applied current of 2.4 and 1.0 hours respectively. These tests will be described in more detail in subsequent reports.

D. PROTECTIVE COATINGS

The fourth objective of the present program is to evaluate the effectiveness and applicability of surface protection in preventing stress-corrosion cracking in 18%-nickel maraging steel. Three coating systems, which

have shown some degree of effectiveness in preventing stress corrosion cracking of H-11 steel are being evaluated on maraging steel. Each of the three coatings offers a different means of protection. The polyurethane coating forms a dense barrier between the environment and the metal. The inorganic zinc coating serves to provide cathodic protection to the metal, while the inhibited epoxy coating protects the metal both by forming a barrier and by the inhibitory action of chromate compounds within the coating. As shown in Table 5 the inhibited epoxy coating is the most effective of the three coatings under our test conditions.

TABLE 1

MILL-CERTIFIED CHEMICAL ANALYSIS OF PROGRAM MATERIALS

Supplier	Heat No.	Composition, % C	Mn	P	S	Si	Ni	Co	Mo	Al	Cr	Zr	Ti	Ca	B	V
*(a) Maraging Steel from Previous Program																
Republic Steel	3960502	0.02	0.08	0.007	0.006	0.15	18.48	7.00	4.84	0.21	0.10	0.035	0.50	-	0.0036	-
Allegheny-Ludlum	448	0.029	0.002	0.004	0.008	0.009	18.51	8.48	4.92	0.089	-	-	0.52	-	-	-
Allegheny-Ludlum	W-24178	0.012	0.01	0.003	0.005	0.01	18.69	8.90	4.92	0.029	-	0.003	0.62	0.006	0.002	-
Allegheny-Ludlum	476	0.02	0.08	0.006	0.005	0.014	18.60	9.05	4.90	0.078	-	-	1.00	-	-	-
Allegheny-Ludlum	W-24254	0.009	0.09	0.002	0.005	0.06	20.41	-	-	0.29	-	0.002	1.40	0.004	0.003	-
(b) Maraging Steel for Present Program																
Republic Steel	3960523	0.029	0.06	0.005	0.006	0.05	17.79	8.50	3.48	0.13	-	-	0.23	-	-	-
Vanadium Alloys	07868	0.02	0.09	0.004	0.005	0.10	17.75	7.60	4.60	0.08	-	0.017	0.52	0.05	0.004	-
Latrobe Steel	C56858	0.03	0.03	0.004	0.008	0.05	18.34	8.00	4.75	0.11	-	0.03	0.49	-	0.004	-
Vanadium Alloys	07268	0.03	0.05	0.004	0.006	0.04	18.54	9.06	4.88	0.09	-	0.088	0.55	0.02	0.003	-
(c) Conventional High-Strength Steels																
Vanadium Alloys	07914	0.38	0.21	0.010	0.008	0.92	-	-	1.33	-	4.75	-	-	-	-	0.51
Allegheny-Ludlum	W-23217	0.495	0.62	0.009	0.003	0.20	0.57	-	0.94	-	1.00	-	-	-	-	0.05

*Some material from previous program will be used to obtain supplementary data.

TABLE 2

MECHANICAL PROPERTIES OF PROGRAM MATERIALS

Code No.	Heat No.	Prior Condition	Heat Treatment Temp, °F	Heat Treatment Hold, hours	0.2% Offset Yield Strength ksi	Ultimate Tensile Strength	Elongation %	Reduction in Area %	R_c Hardness	ASTM Grain Size No.	Crack Growth Energy (G_c) $lb/in.^2$
H-1	W-24254	Annealed*	850	4	291.3	302.2	3	17	54	15	59
H-2	↓	50% CR	↓	↓	321.0	327.1	3	25	55-1/2	-	22.9
H-3	↓	75% CR	↓	↓	298.3	308.8	2-1/2	13	55	-	15.8
H-W	W-24254	Welded	850	4	245.0	252.0	1-1/2	5	-	-	-
I-5	477	50% CR	900	3	278.0	280.7	2	8	51-1/2	-	485
I-4	3960502	Annealed	↓	↓	249.9	254.7	4	37	50-1/2	12	825
I-2	↓	50% CR	↓	↓	302.5	308.1	4	26	52-1/2	-	343
I-W	3960502	Welded	↓	↓	236.6	242.0	2	20	-	-	-
I-6	448	Annealed	↓	↓	255.4	265.9	5	9	52	13-1/2	760
I-7	448	50% CR	↓	↓	331.0	332.5	1-1/2	7	55	-	(573)
I-3	W-24178	Annealed**	↓	↓	283.0	294.0	8	38	53-1/2	-	625
I-1	W-24178	50% CR	↓	↓	323.8	328.4	1-1/2	28	55	-	228
I-8	476	Annealed	↓	↓	323.3	330.0	2-1/2	27	56	14-1/2	430
I-9	476	50% CR	900	3	354.4	354.9	1	1-1/2	58	-	159
K	3960523	Annealed	900	3	181.5	190.7	5	43	42	8	1020
L	07868	↓	↓	↓	248.2	(248.2)	4	-	49	12-1/2	857
M	C56858	↓	↓	↓	269.7	275.7	5	34	51-1/2	7-1/2	755
N	07268	Annealed	900	3	279.1	288.1	4	18	52	12-1/2	639
A-4	07914	1 hr, 1850°F, OQ***	1075	4 + 4	219.2	257.7	7	44	49	-	-
A-3	↓	↓	1025	↓	232.6	284.8	6	42	52-1/2	-	-
A-2	↓	↓	975	↓	223.5	280.6	6-1/2	43	53	-	46.0
A-1	07914	1 hr, 1850°F, OQ	925	4 + 4	222.2	292.4	7	40	54	-	33.5
B-4	W-23217	40 min, 1550°F, OQ	1100	2	203.1	218.5	11	46	44	-	785
B-3	↓	↓	900	↓	204.6	226.4	7-1/2	43-1/2	44	-	446
B-2	↓	↓	800	↓	214.5	241.2	7	38	45-1/2	-	374
B-1	W-23217	40 min, 1550°F, OQ	600	2	237.4	281.3	6	25	51-1/2	-	192

NOTES: *CR - Cold reduced at mill
**Received cold reduced, annealed 1 hour at 1500°F, air cooled.
***OQ = Oil Quenched.

TABLE 3

BENT-BEAM STRESS-CORROSION TEST RESULTS

Table 2 Code No.	Heat No.	0.2% Offset Yield Strength	Aerated Distilled Water Ratio	Aerated Distilled Water Time	Aerated 3% NaCl Solution Ratio	Aerated 3% NaCl Solution Time	120°F Distilled Water Ratio	120°F Distilled Water Time	140°F Water-Saturated Air Ratio	140°F Water-Saturated Air Time	160°F Distilled Water Ratio	160°F Distilled Water Time
			Failure Ratio* and Failure Time, Hours,** in Various Environments									
(a) Maraging Steel from Previous Program												
(1) Mill annealed												
H-1	W-24254	291.3	3/3	10.5	3/3	7.8	0/0	-	3/3	105	2/2	47
I-4	3960502	249.9	3/3	70	3/3	140	3/3	164	3/3	475	3/3	138
I-6	448	255.4	1/3	NF 3600	0/2	NF 3600	0/0	-	3/3	266	1/1	355
I-1	W-24178	283.0	3/3	36	3/3	36	2/2	145	3/3	21.5	2/2	16
I-8	476	323.3	3/3	24	2/2	6.5	1/1	120	3/3	72	0/0	-
(2) Welded												
H-W	W-24254	245.0	3/3	47	3/3	0.8	2/2	42	3/3	14	2/2	1.5
I-W	3960502	236.6	3/3	360	3/3	115	2/2	148	3/3	139	2/2	31
(3) 50% Cold-Reduced												
H-2	W-24254	321.0	1/3	NF 2900	0/3	NF 2900	2/2	810	3/3	5800	2/2	138
I-5	477	278.0	0/3	NF 3600	0/2	NF 3600	0/0	-	2/3	9800	1/1	1010
I-2	3960502	302.5	3/3	1780	2/3	3200	2/2	256	3/3	380	2/2	150
I-7	448	331.0	0/3	NF 3600	0/2	NF 3600	0/1	NF 1000	3/3	3080	1/1	690
I-3	W-24178	323.0	4/4	536	3/3	1580	2/2	473	3/3	290	2/2	186
I-9	476	354.4	3/3	4460	2/2	20	1/1	304	3/3	930	1/1	136
(b) Maraging Steel from Present Program												
K	3960523	181.5	0/3	NF 3880	0/3	NF 3880	1/2	NF 3880	3/3	1560	3/3	740
L	07868	248.2	3/3	1032	0/3	NF 3170	3/3	596	3/3	578	3/3	300
M	C56858	269.7	0/3	NF 2520	3/3	1940	3/3	324	3/3	188	3/3	138
N	07268	279.1	3/3	510	3/3	119	3/3	336	3/3	535	3/3	138
(c) Conventional High Strength Steels												
A-4	07914	219.2	1/2	1745	2/2	853	2/2	784	0/2	NF 1680	2/2	584
A-3	↓	232.6	2/2	544	2/2	416	2/2	198	1/2	548	2/2	196
A-2		223.5	2/2	294	2/2	179	2/2	138	2/2	166	2/2	150
A-1	07914	222.2	3/3	143	3/3	18	4/4	82	3/3	482	2/2	73
B-4	W-23217	203.1	0/3	NF 2800	0/2	NF 2800	0/2	NF 2800	0/3	NF 2800	0/2	NF 1980
B-3	↓	204.6	2/3	2300	0/3	NF 2800	2/3	2825	3/3	908	2/2	500
B-2		214.5	3/3	1200	0/3	NF 2800	3/3	700	3/3	509	2/2	198
B-1	W-23217	237.4	3/3	720	1/3	NF 2800	3/3	213	3/3	141	2/2	105

NOTES: All specimens stressed to 75% of yield strength.

* Ratio of samples failed to samples tested.

** Median failure times, in hours, for three specimens, average for two specimens.

TABLE 4

U-BEND STRESS-CORROSION TEST RESULTS

Table 2 Code No.	Heat No.	0.2% Offset Yield Strength	Failure Ratio* and Failure Time, Hours,** in Various Environments: Aerated Distilled Water Ratio	Aerated Distilled Water Time	Aerated 3% NaCl Solution Ratio	Aerated 3% NaCl Solution Time	120°F Distilled Water Ratio	120°F Distilled Water Time	140°F Water-Saturated Air Ratio	140°F Water-Saturated Air Time	160°F Distilled Water Ratio	160°F Distilled Water Time
(a) Maraging Steel from Previous Program												
H-1	W-24254	291.3	2/2	3.4	2/2	2.4	3/3	194	0/0	--	3/3	46
I-4	3960502	249.9	2/2	825	0/0	--	2/2	133	2/2	167	3/3	20
(b) Maraging Steel from Present Program												
K	3960523	181.5	3/3	2140	1/3	NF 4150	3/3	333	4/4	240	3/3	130
L	07868	248.2	3/3	620	3/3	1776	3/3	191	3/3	167	3/3	40
M	C56858	269.7	3/3	430	3/3	1174	3/3	237	3/3	70	3/3	20
N	07268	279.1	2/3	1823	1/3	NF 4440	3/3	410	3/3	470	2/2	35
(c) Conventional High Strength Steels												
A-4	07914	219.2	2/2	510	2/2	267	2/2	280	2/2	500	2/2	330
A-3	↓	232.6	2/2	174	2/2	232	2/2	172	2/2	30	1/1	232
A-2	↓	223.5	2/2	200	2/2	143	2/2	119	2/2	70	2/2	144
A-1	07914	222.2	2/2	82	2/2	24	2/2	70	2/2	24	2/2	51
B-4	W-23217	203.1	0/2	NF 3000	0/2	NF 3000	0/2	NF 3000	0/2	NF 3000	0/1	NF 3000
B-3	↓	204.6	2/2	1470	0/2	NF 3000	2/2	974	2/2	372	1/1	620
B-2	↓	214.5	2/2	144	2/2	2600	2/2	537	2/2	217	1/1	380
B-1	W-23217	237.4	2/2	4.9	2/2	1.0	2/2	2.4	2/2	<1	1/1	<1

Notes: All specimens stressed beyond yield strength.

*Ratio of samples failed to samples tested.

**Median failure times for three specimens, average for two specimens.

TABLE 5

BENT BEAM STRESS CORROSION TESTS FOR COATINGS EVALUATION

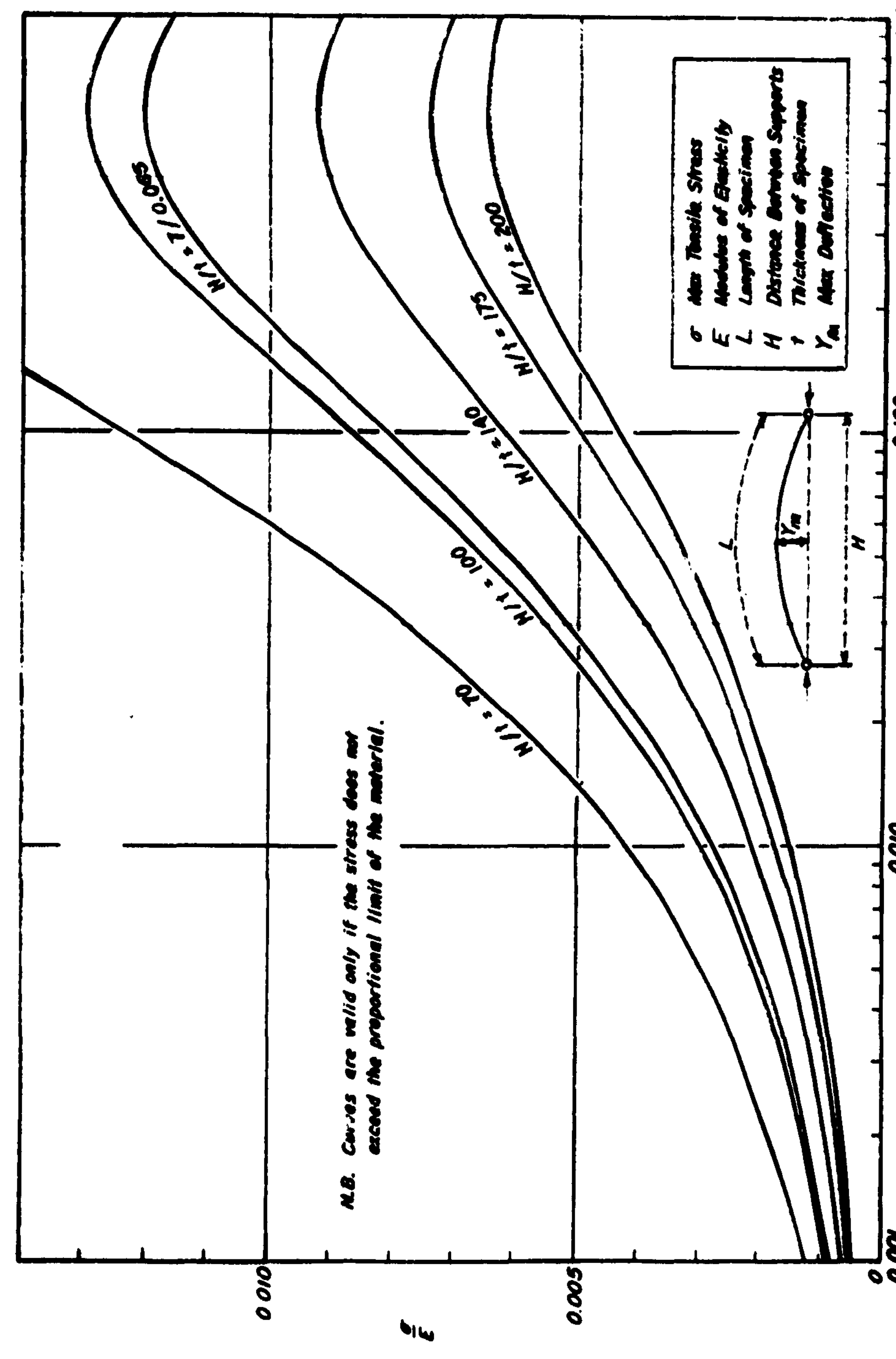
		Aerated 3% NaCl Solution		140°F Water-Saturated Air	
Base Metal	Coating	Failure Ratio*	Median Failure Time hours	Failure Ratio*	Median Failure Time hours
H-11 Steel	None	4/4	1.5	2/2	64
	Polyurethane	3/3	1380	6/6	3500
	Inorganic zinc	2/2	687	2/2	821
H-11 Steel	Inhibited epoxy	0/2	NF 3100	3/3	2720
18% Ni Maraging Steel	None	3/3	119	3/3	535
	Polyurethane	0/3	NF 3880	3/3	1560
	Inorganic zinc	3/3	288	3/3	140
18% Ni Maraging Steel	Inhibited epoxy	0/3	NF 3880	2/3	1870

*Ratio of samples failed to samples exposed.

1062-616

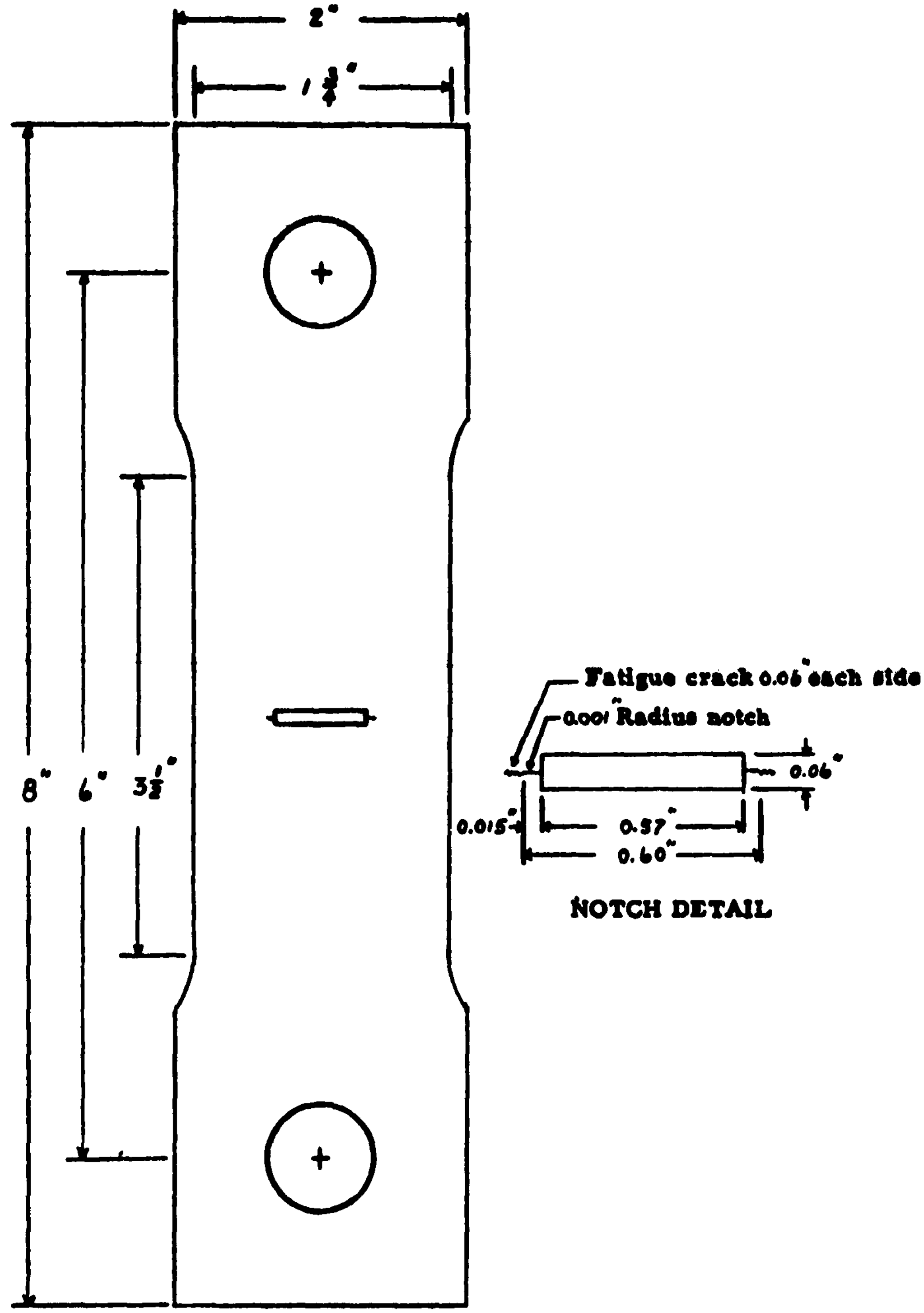
Bent-Beam Test Fixture and Specimens

Figure 1



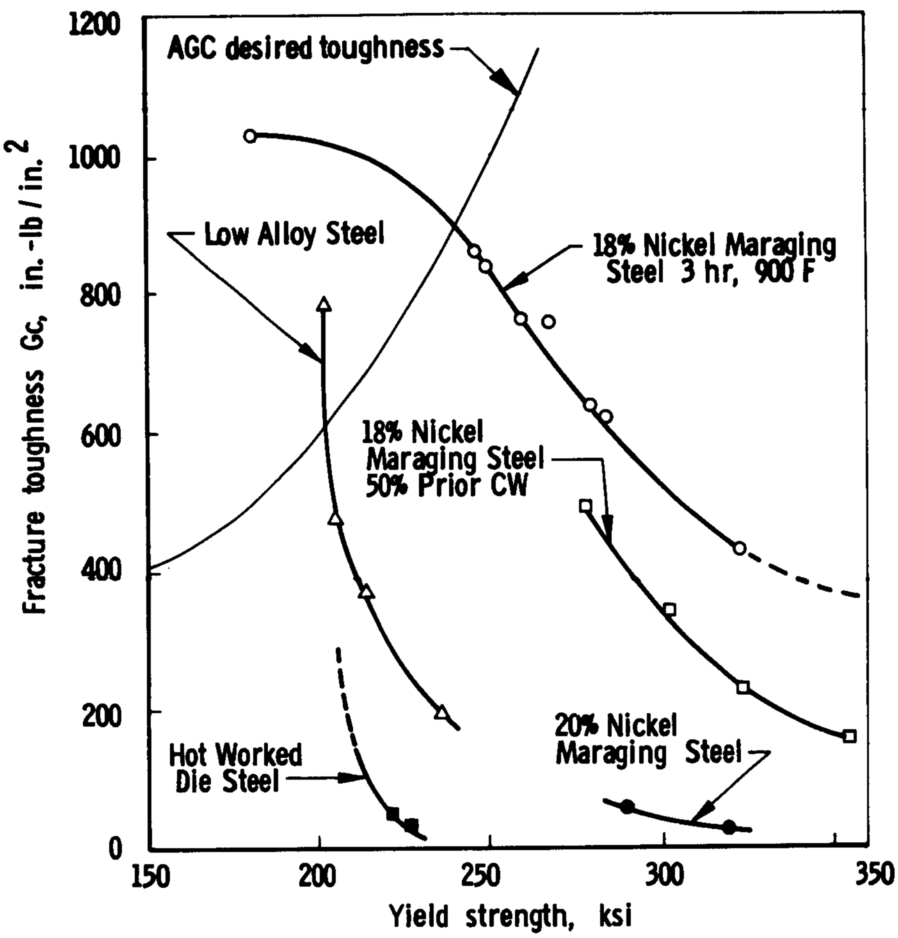
Beam Length-Stress Relationship

Figure 2

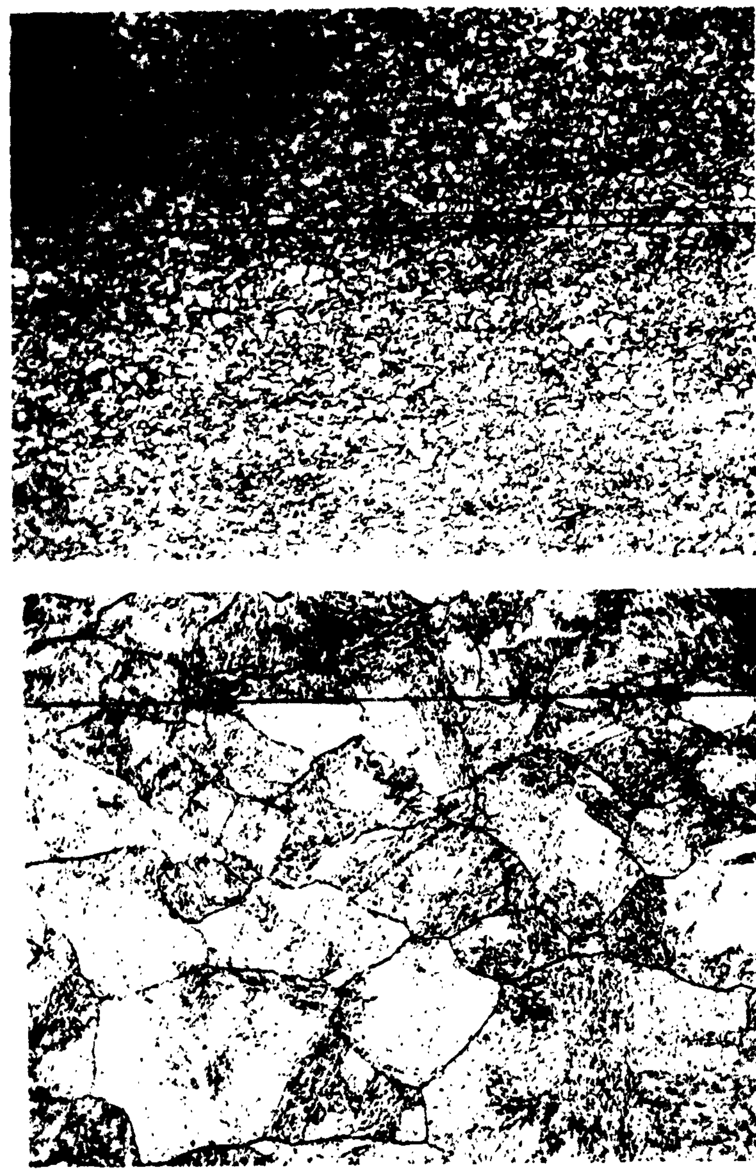


Center-Notch Specimen Configuration

Figure 3



FRACTURE TOUGHNESS VALUES
CENTER NOTCHED SPECIMENS



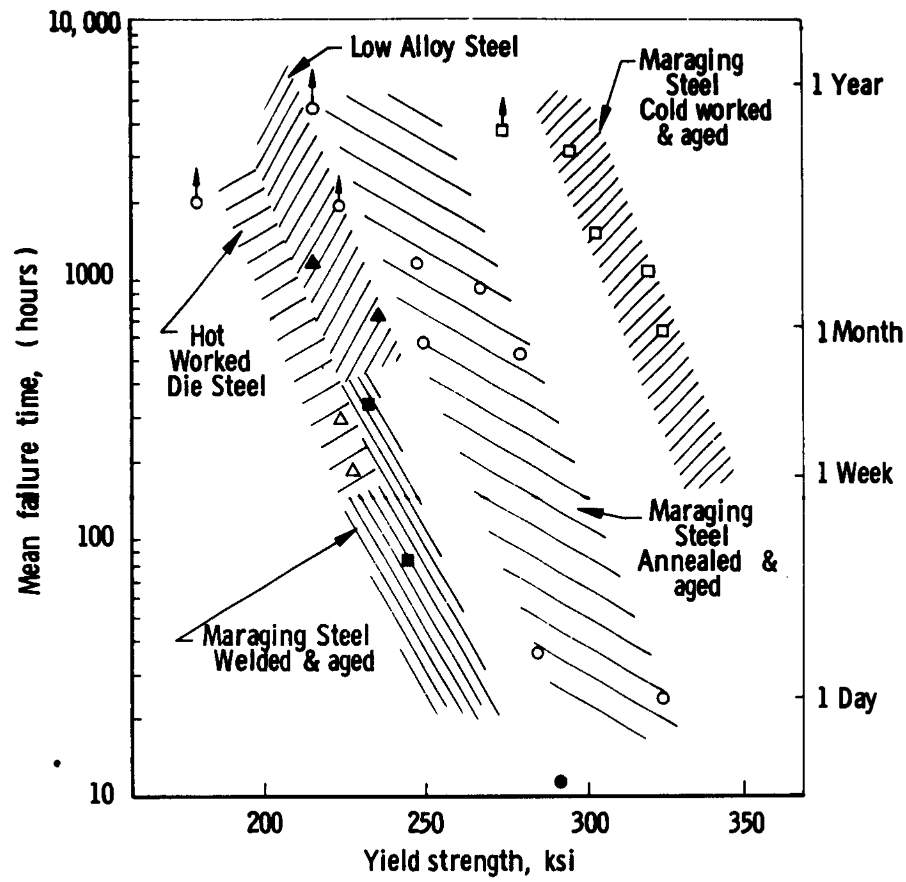
Grain Size Variations Encountered in 18% Nickel Maraging Steels

(Top) Heat 476 (Bottom) Heat 3960523

Etchant: 5% chromic acid, electrolytic

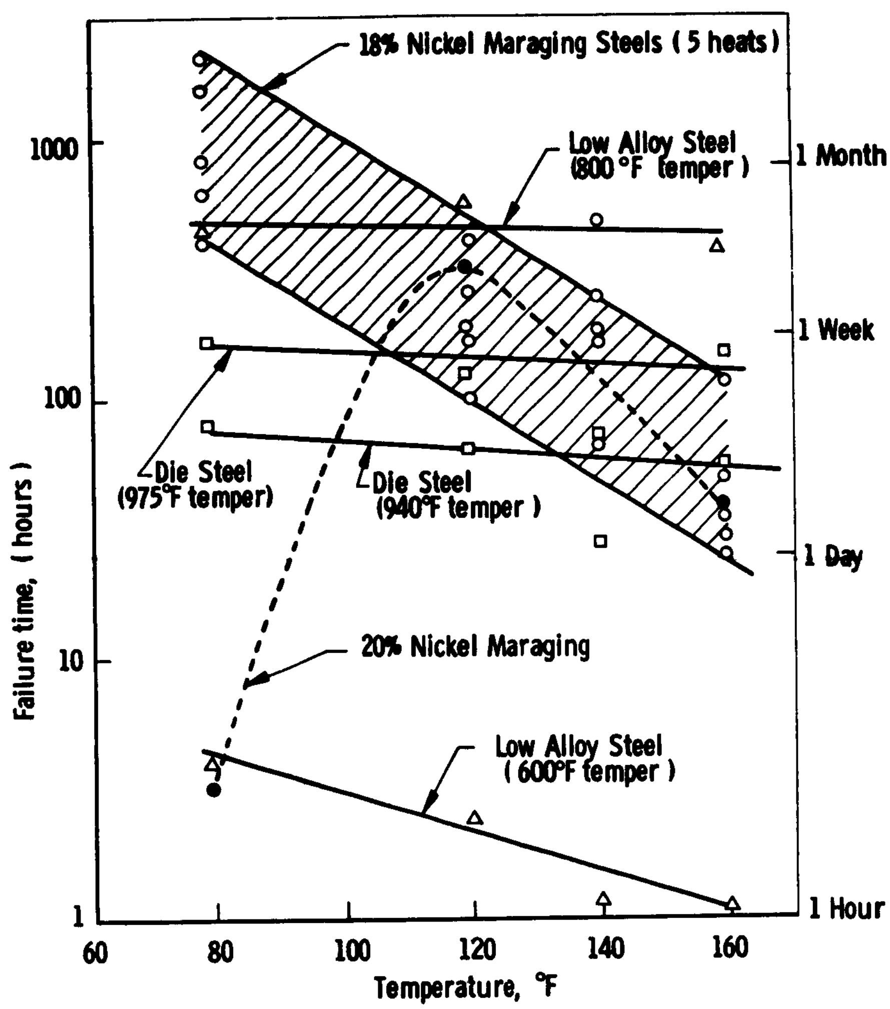
Magnification: 500X

Figure 5



AERATED DISTILLED WATER - BENT BEAM TESTS

Figure 6



EFFECT OF TEMPERATURE IN DISTILLED WATER
U-BEND TESTS

Figure 7

Typical Crack in 18% Nickel Maraging Steels

Heat 3960523

Etchant: Dilute marbles

Magnification: 1400X

Figure 8

Typical Cracks in Conventional High-Strength Steels

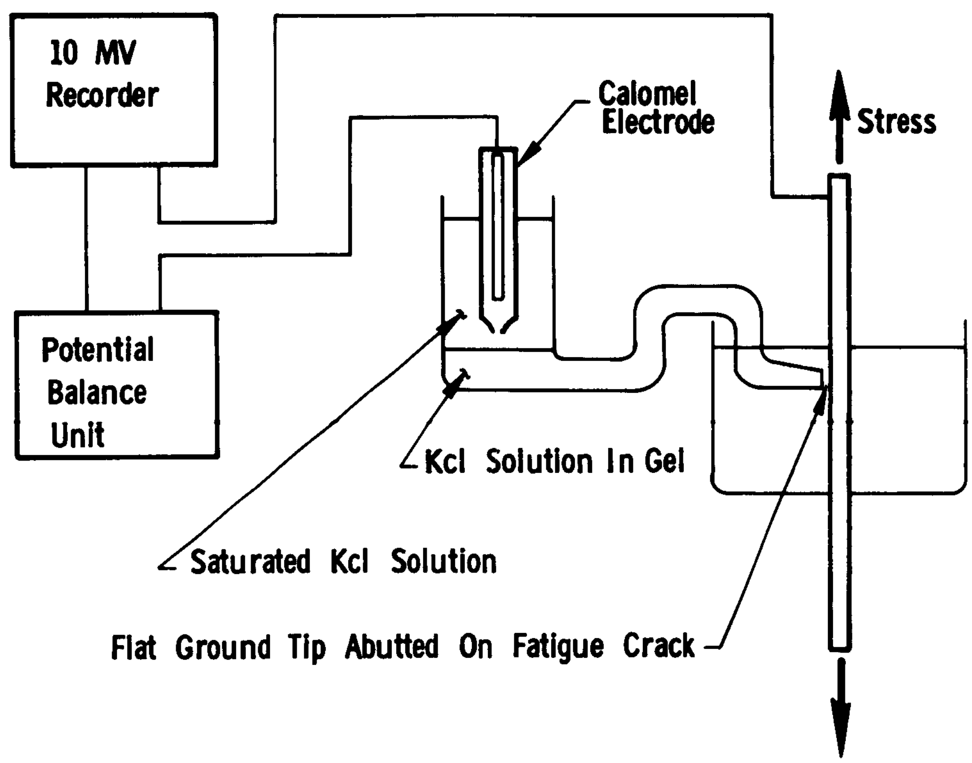
(Top) Low alloy steel Heat W-23217

(Bottom) Hot worked die steel Heat 07914

Etchant: Hcl picral

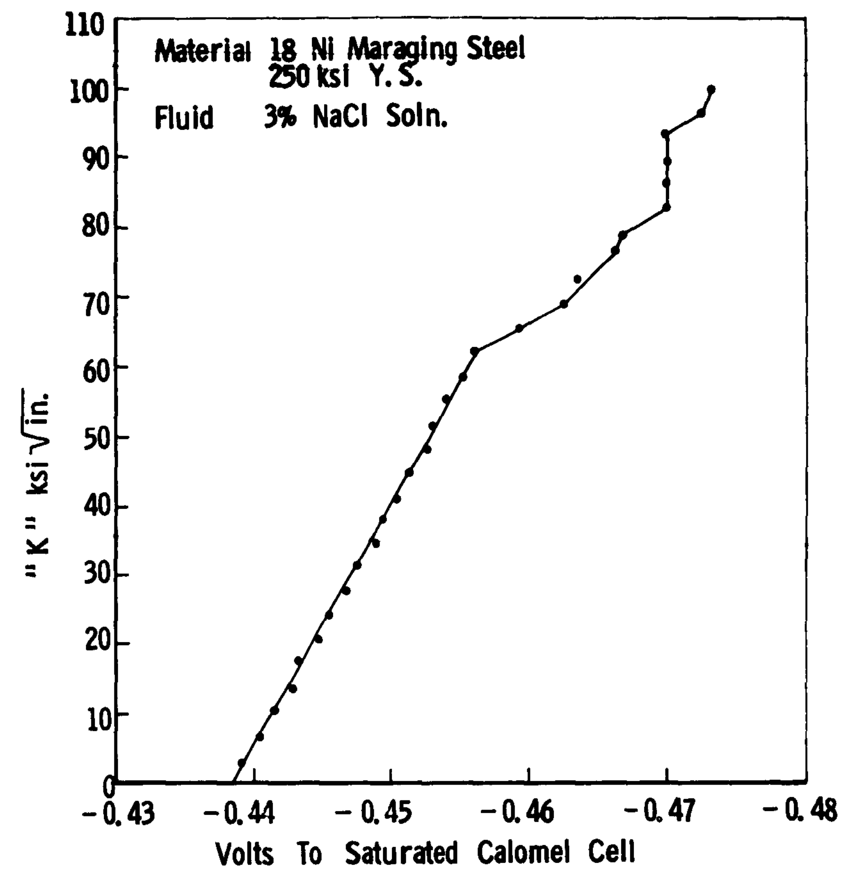
Magnification: 500X

Figure 9



STRESS-CORROSION POTENTIAL MEASURING APPARATUS

Figure 10



EFFECT OF STRESS FIELD PARAMETER, K, ON CRACK-TIP CORROSION POTENTIAL

Figure 11

(Top) Experimental Test for the Determination of Applied Potential on Stress-Corrosion Cracking

(Bottom) Test Cell Showing Specimen and Auxiliary Platinum Electrode

Figure 12